AD 727 669

TECHNICAL REPORT
71-34-FL

STUDY OF FAT STABILITY AND SHELF LIFE OF CANNED MARGARINE

by

P. K. Jarvi, C.A. Overley
and
J. W. Robertson

Swift and Company,
Research and Development Center,
Oak Brook, Illinois 60521

Contract No. DAAG 17-69-C-0201

March 1971

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760

U.S. ARMY MATERIEL COMMAND

Food Laboratory
FL-125

AD 727 669

AD___________

TECHNICAL REPORT
71-34-FL

STUDY OF FAT STABILITY AND SHELF LIFE OF CANNED MARGARINE

by

P. K. Jarvi, C. A. Overley and J. W. Robertson

Swift and Company
Research and Development Center
Oak Brook, Illinois 60521

Contract No. DAAG 17-69-C-0201

Project reference:
Production Engineering

Series: FL-125

March 1971

Food Laboratory
U. S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01760

13. Abstract (Continued):

a. The peroxide value

b. The AOM stability

c. Response of the flavor panel as a function of AOM and PV values

d. Organoleptic responses of the panel as a function of temperature, time, PV, and AOM values.

It was found that the stability of the canned margarine is mainly and predictably dependent upon the PV and AOM value of the stock oil. Also, a close relationship exists between the PV's of the stock oil and of the oil separated from the canned margarine by thermal demulsification. However, no useful relationship for predicting the shelf life from the AOM values of the oil separated from the canned margarine by the four methods could be established.

It is evident that the autoxidation of the margarine fat does not proceed in the sealed container, but the hydroperoxides present originally will decompose and cause some deterioration and development of the off flavor and odor and fading of the color.

The AOM value of the separated oil is not well suited for judging the shelf life of canned margarine because the AOM test has been designed to predict the stability in the presence of oxygen.

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Swift and Company Research and Development Center Oak Brook, Illinois 60521	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

STUDY OF FAT STABILITY AND SHELF LIFE OF CANNED MARGARINE

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final December 1969 - December 1970

5. AUTHOR(S) (First name, middle initial, last name)

P. K. Jarvi, C. A. Overley and J. W. Robertson

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
March 1971	31	0

8a. CONTRACT OR GRANT NO. DAAG 17-69-C-0201 b. PROJECT NO. Production Engineering c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) 71-34-FL FL-125

10. DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY U.S. Army Natick Laboratories Natick, Massachusetts 01760

13. ABSTRACT

The aim of the study of canned margarine was to determine the relationships between the keeping quality (shelf life) and the Active Oxygen Method (AOM) fat stability test and Peroxide Value (PV) made on the oil incorporated into the margarine and separated from it. Methods of oil separation were also studied.

Five margarines were formulated containing varying amounts of oxidized oil (0, 3.1, 5.8, 12.4, 20%) packed in sealed cans and stored at +100°F and -20°F. Initial and monthly samples for a period of six months were subjected to four methods of oil separation; thermal demulsification, solvent extraction, vacuum distillation, and chemical demulsification. The separated oils were evaluated from AOM and PV. The margarines themselves were sensory evaluated for color, odor, flavor and mouth feel. Statistical procedures were used for evaluation of the results.

A method for oil separation (thermal demulsification) was selected as basis of statistical evaluation, because it allows the most accurate prediction of the organoleptic responses: odor, color, and off flavor, as a function of the percent of oxidized oil, AOM, and PV.

The results of the stability tests were evaluated statistically. Equations were derived for predicting:

(See continuation sheet)

DD FORM 1 NOV 65 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified
Security Classification

Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Stability	8					
Fats	9					
Margarine	5					
Tests	8		8		1	
Active oxygen method (AOM)	10		8			
Peroxide value (PV)	10		8			
Oil			1		2	
Separation					8	
Storage stability			4			

Unclassified
Security Classification

Approved for public release;
distribution unlimited.

AD______

TECHNICAL REPORT
71-34-FL

STUDY OF FAT STABILITY AND SHELF LIFE OF CANNED MARGARINE

by

P. K. Jarvi, C. A. Overley and J. W. Robertson

Swift and Company
Research and Development Center
Oak Brook, Illinois 60521

Contract No. DAAG 17-69-C-0201

Project reference:
Production Engineering

Series: FL-125

March 1971

Food Laboratory
U. S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01760

FOREWORD

Increasing reliance upon end-item inspection in lieu of inspection of ingredient materials in Armed Forces procurement necessitates reconsideration of test procedures for specification enforcement. The shelf-life of canned margarine, the most widely used spread in operational rations, had been controlled by a stability requirement on the base oil stock. This requirement was enforced by testing the oil stock with the Active Oxygen Method (AOM) stability test. The validity of this test applied to the end-item, or more exactly to the oil phase separated from the finished margarine has been challenged. Two objections were made: (1) that methods used to separate the oil phase were imperfect and (2) that the AOM stability of the seperated oil reflects neither the stability of the base oil nor that of the margerine. It was important, therefore, to determine the validity of these objections.

The work covered in this report, performed at the Research and Development Center of Swift and Company under Contract Number DAAG 17-69-C-0201 (December 1969 - December 1970) represents an investigation (1) of the effects of methods of oil seperation on the AOM stability of the separated oil, (2) the relationships between the AOM's of the base oil stocks and of the separated oil, and (3) the relationships between these two AOM values and the shelf-life of margarine. The investigators were F. K. Jarvi, C. A. Overley and J. W. Robertson.

The U. S. Army Natick Laboratories Project Officer was A. S. Henick and the Alternate Project Officer was I. Bloch, both of the Food Chemistry Division, Food Laboratory.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

The aim of the study of canned margarine was to determine the relationships between the keeping quality (shelf life) and the Active Oxygen Method (AOM) fat stability test and Peroxide Value (PV) made on the oil incorporated into the margarine and separated from it. Methods of oil separation were also studied.

Five margarines were formulated containing varying amounts of oxidized oil (0, 3.1, 5.8, 12.4, 20%) packed in sealed cans and stored at +100°F. and -20°F. Initial and monthly samples for a period of six months were subjected to four methods of oil separation; thermal demulsification, solvent extraction, vacuum distillation, and chemical demulsification. The separated oils were evaluated for AOM and PV. The margarines themselves were sensory evaluated for color, odor, flavor and mouth feel. Statistical procedures were used for evaluation of the results.

A method for oil separation (thermal demulsification) was selected as basis of statistical evaluation, because it allows the most accurate prediction of the organoleptic responses: odor, color, and off flavor, as a function of the percent of oxidized oil, AOM, and PV.

The results of the stability tests were evaluated statistically. Equations were derived for predicting:

a. The peroxide value

b. The AOM stability

c. Response of the flavor panel as a function of AOM and PV values

d. Organoleptic responses of the panel as a function of temperature, time, PV, and AOM values.

It was found that the stability of the canned margarine is mainly and predictably dependent upon the PV and AOM value of the stock oil. Also, a close relationship exists between the PV's of the stock oil and of the oil separated from the canned margarine by thermal demulsification. However, no useful relationship for predicting the shelf life from the AOM values of the oil separated from the canned margarine by the four methods could be established.

It is evident that the autoxidation of the margarine fat does not proceed in the sealed container, but the hydroperoxides present originally will decompose and cause some deterioration and development of the off flavor and odor and fading of the color.

The AOM value of the separated oil is not well suited for judging the shelf life of canned margarine because the AOM test has been designed to predict the stability in the presence of oxygen.

INTRODUCTION

The study of fat stability and shelf life of canned margarine involves the relationships between keeping quality (shelf life) and the Active Oxygen Method (AOM) fat stability test and Peroxide Value (PV) made on the oil incorporated into the margarine and separated from it.

The problem here is the same as is usually encountered with the stability of fats. The autoxidation and the decomposition of the hydroperoxide are usually described as a chain reaction:

DECOMPOSITION OF HYDROPEROXIDE (IMITATION)

$$ROOH \longrightarrow RO^\circ + HO^\circ$$

CHAIN PROPAGATION

$$R^\circ + O_2{}^\circ \xrightarrow{k_2} RO_2{}^\circ$$

$$RO_2{}^\circ + RH \xrightarrow{k_3} ROOH + R^\circ$$

CHAIN TERMINATION

$$\left.\begin{array}{l} RO_2{}^\circ + RO_2{}^\circ \xrightarrow{k_6} \\ RO_2{}^\circ + R^\circ \xrightarrow{k_5} \\ R^\circ + R^\circ \xrightarrow{k_4} \end{array}\right\} - \text{PRODUCTS}$$

Among the side products of the reaction are formed the off flavor and odor causing compounds.

The results of all examinations were evaluated by statistical procedures for analysis of variance and co-variance, and differences after storage were correlated with the results of the initial analyses and tests, both on the oil ingredients and on the oils separated from the finished products. Regression equations, together with standard deviations were developed to permit the prediction of shelf life of canned margarine from the results.

MATERIALS AND METHODS

Oils

Refined and bleached cottonseed oil used as the base oil was hardened to meet the solid fat index values as required in Mil M-10958D Military Specification, Canned Margarine Section 3.4.1. Results are shown in Table I.

TABLE I

INITIAL OIL

SOLID FAT INDEX VALUES (SFI)

Temp. (°F)	Specifications	Lot No. 1	2	3	4	5
50	28.0-32.0	30.7	30.7	30.7	31.8	31.9
70	18.0-21.0	21.2	21.2	21.2	22.2	21.2
80	17.5-20.5	18.7	18.7	18.7	19.7	18.3
92	11.5-13.0	11.9	11.9	11.9	12.4	11.4
100	7.5-9.5					
105	5.5-8.0	5.8	5.8	5.8	5.8	5.5

In order to obtain fat stability tests ranging from 40 to 80 hours, a preoxidized oil was used. This was prepared by segregating a portion of the initial hardened oil, rapidly mixing to form a vortex with air and keeping at a temperature of 170-180°F. After 237 hours mixing and having a peroxide value of 45.0, this oxidized oil was added to the initial oil test batches in amounts from 0 to 20%, from which the margarines were prepared, see Table II.

TABLE II

OIL BLENDS

	Lot 1	Lot 2	Lot 3	Lot 4	Lot 5
% Oxidized Oil	20.0	12.4	5.8	3.1	-
Peroxide Value	15.2	11.0	7.6	2.6	2.4
AOM (hours)	64	76	87	121	96

The analytical requirements as indicated in Section 3.4.1 were followed except for peroxide value and fat stability. Artificial flavor was omitted to enable a better following of the development of off flavors. Analysis of the initial samples of the lots prepared are as seen in Table III.

TABLE III

ANALYSES OF FINISHED MARGARINE

Lot	Moisture	Fat	Curd	Salt
1	11.3	84.4	1.0	3.3
2	12.7	82.9	1.2	3.2
3	11.8	83.9	1.0	3.3
4	10.9	84.7	1.4	3.0
5	12.6	82.0	1.7	3.7

The samples of margarine were packed in size 603 by 700 cans, and each lot put at -20°F. and 100°F. for aging. Duplicate samples were submitted to the Natick Laboratories.

AOM and PV

The AOM and PV determinations were made by using the official methods of the American Oil Chemists Society.

METHODS OF OIL SEPARATION

Since the PV and AOM's must be run on oil separated from the finished margarine, a study was made to find the best means of separating the oil, with the idea of improving the correlation between the keeping quality of the margarine versus the fat stability and the peroxide value tests.

The following methods of separation were investigated: thermal demulsification, solvent extraction, evaporation under vacuum, chemical demulsification.

Thermal Demulsification:

Place 400 grams of margarine in a 600 ml beaker and hold at 150-160°F. for 16 hours (overnight). Decant the separated oil through a coarse filter paper held in an oven at 150-160°F., (Eaton-Dikeman No. 617 or equivalent) into a container.

Solvent Extraction:

Place 200 grams of margarine and 400 cc Skellysolve F in an 800 cc beaker. Stir by hand at room temperature until well mixed and heat up at the same time to 95°F. Allow to stand for a minimum of ten minutes, decant through a coarse filter paper (Eaton-Dikeman 617 or equivalent) into a round bottom flask of 2-3000 ml capacity. Evaporate under vacuum (water aspiration) in water bath of approximately 180°F. minimum. Last traces of solvent are removed by means of a mechanical pump with acetone-dry ice trap.

Evaporation:

Place 200 grams of margarine in a 500 ml round bottom, ground neck flask. Melt and heat half submerged in steam bath for 2-2 1/2 hours under vacuum of 25 mm Hg or less using a Rinco rotary evaporator. When all moisture has been removed, filter while hot using suction through a warm Beuchner funnel (Eaton-Dikeman No. 617 paper or equivalent) to a container.

Chemical Demulsification:

Place 200 grams of margarine in a 600 ml beaker, melt on steam bath and add 300 ml of 30% hydrochloric acid. Continue to heat on steam bath for three hours with occasional stirring with a glass rod. When fat has separated, decant through coarse filter paper (Eaton-Dikeman No. 617 or equivalent) to a container.

SENSORY EVALUATION

Samples of each lot of margarine held at -20°F. and +100°F. were removed monthly for six months. The margarines were sensory evaluated for color, odor, flavor, and mouth feel by professional panelists using as control a sample containing 0% oxidized oil and held at -20°F. The panelists applied the following scale in their evaluation.

Color	1.00 = No difference	6.00 = Very much difference
Odor	1.00 = No difference	6.00 = Very much difference
Off-Flavor	1.00 = No difference	6.00 = Very much difference
Mouth Feel	1.00 = Poor	4.00 = Excellent

RESULTS AND DISCUSSION

General

The selection of a method of oil separation was based on results obtained through step-wise multiple linear regressions.* In the regression AOM, peroxide values and the interaction (AOM by peroxide value) were regressed with the organoleptic responses. Regressions were executed for each of the four methods of oil extraction, in an effort to predict the organoleptic responses.

The thermal demulsification method was selected as the most sensitive method of oil extraction, since it allows significant ($\alpha = .05$) prediction of all organoleptic responses. Further examination of methods, revealed smaller standard errors (S.E.) and larger percents of included variability in the thermal demulsification method.

Table IV shows the standard errors (S.E.) and the percent included variability (R^2), as related to the AOM and peroxide values using four methods of oil extraction.

TABLE IV

SELECTION OF OIL EXTRACTION METHOD

	Color		Odor		Off-Flavor	
	S.E.	R^2	S.E.	R^2	S.E.	R^2
Thermal AOM	1.2	10	+	+	+	+
PV	1.1	23	.6	9	.7	22
AOM & PV	1.1	23	.6	9	.7	22
Solvent AOM	+	+	+	+	.8	08
PV	1.2	12	+	+	.8	10
AOM & PV	1.2	12	+	+	.7	22
Vacuum AOM	+	+	+	+	+	+
PV	1.2	12	+	+	.7	15
AOM & PV	1.2	12	.7	6	.7	15
Chem. AOM	+	+	+	+	+	+
PV	1.2	7	+	+	.7	12
AOM & PV	1.2	7	+	+	.7	12

\+ Not predictable ($\alpha = .05$)

* N. R. Draper and H. Smith, "Applied Regression Analysis". John Whiley and Son, Inc., New York, N. Y., 1966.

The development of rancidity in the margarines stored at 100°F. and -20°F. as measured by the PV may be seen in Figures 1 and 2 and Table V.

The AOM (fat stability) results are presented in Figures 3 and 4, and Table VI.

The sensory evaluation results of the margarine have been collected in Figures 5, 6, 7, 8, 9, 10 and Table VII.

All of the correlation coefficients for the variables studied are presented in Table VIII. It is our general experience that correlation coefficients between a physical/chemical test and an organoleptic result should be on the order of 0.75 or higher to have any practical (utilitarian) value. However, since statistical significance exists above 0.23 some useful conclusions can be made.

It is evident that the thermal peroxide value correlates significantly with the peroxide values obtained by the solvent, vacuum and chemical separation procedures. The thermal AOM's also correlate significantly with the AOM's obtained by the solvent and vacuum separation procedures. There is not a significant correlation with the AOM's obtained by the chemical separation.

Off flavor correlates significantly with the peroxide values obtained by all four methods of oil separation. Odor correlates with the thermal peroxide value only.

As can be expected, off flavor and odor show a highly significant correlation. Color shows a significant correlation with off flavor and odor.

The percent of oxidized oil used to control original AOM and PV values shows a highly significant correlation with all the studied variations except vacuum and chemical AOM.

Mouth feel does not correlate significantly with any of the variations studied (did not change much during the study).

Temperature has been a highly significant factor to the development of color, odor and off flavor, but on the other hand, no significant correlation can be found with respect to the PV and AOM values obtained by any of the methods tested. An explanation may be found in that the margarine was kept in sealed cans, with no possibility for additional oxygen to get to the product, so that the oxidation reaction could not proceed, and new hydroperoxides were not formed. However, the increase of off flavor and odor obviously resulted from the decomposition of the hydroperoxides present in the product at the time of the sealing of the cans.

REGRESSION EQUATIONS

By means of statistical analysis of the data presented in the earlier tables, the following equations predicting peroxide values and AOM values as a function of temperature (°F), time (in days), and percent oxidized oil were obtained.

$$\text{Peroxide Value} = 1.52025 + 52.76306\ (\%\ \text{oxidized oil}) + .00120\ (\text{temperature})^2 - .00143\ (\text{temperature} \times \text{time} \times \%\ \text{oxidized oil}) - .06923\ (\text{temperature}) \pm 1.99272$$

$$\text{AOM Value} = 63.28406 - 546.07771\ (\%\ \text{oxidized oil})^2 + 87.09573\ (\%\ \text{oxidized oil}) \pm 5.82410$$

A set of equations was also developed for canned margarine for predicting the organoleptic responses of the panel (without the time element) as a function of the active oxygen method, peroxide value, or their squares. These equations may be seen below:

ORGANOLEPTIC PREDICTION EQUATIONS

	Equation	Std. Dev. from Means	Std. Dev from Regression	R^2*
Color	=1.5317 + .1457 (PV)	1.2388	1.0961	23
	=21.478 − .5506 (AOM) + .0040 (AOM)^2	1.2388	1.1930	10
Odor	=1.6324 + .0505 (PV)	.6843	.6576	09
Off Flavor	=1.84694 + .0907 (PV)	.7875	7010	22

*The percent of the total variance of the response, explained by the regression equation.

For actual shelf life prediction of canned margarine, this meaning the organoleptic response of a test panel at any given time, equations were formed by using the PV and AOM values of the initial oil and oil separated from the initial margarines. The derived equations are:

1. EQUATIONS PREDICTING ORGANOLEPTIC RESPONSES OF THE PANEL TO CANNED MARGARINE AS A FUNCTION OF TEMPERATURE (°F.), TIME (DAYS), PEROXIDE VALUES AND AOM VALUES

Note: The peroxide values and AOM value were derived from the initial oil before the addition of milk.

$$\begin{aligned}\text{COLOR} = -1.02470 &+ .000004 \text{ (temperature x time x peroxide value)}\\ &+ .00913 \text{ (peroxide value)}^2\\ &+ .00032 \text{ (temperature)}^2\\ &+ .02171 \text{ (AOM value)}\\ &- .01614 \text{ (temperature)}\\ &\pm .80555\end{aligned}$$

$$\begin{aligned}\text{ODOR} = 1.99533 &+ .000003 \text{ (temperature x time x peroxide value)}\\ &+ .00038 \text{ (temperature x peroxide value)}\\ &+ .00006 \text{ (time)}^2\\ &- .01077 \text{ (time)}\\ &\pm .48004\end{aligned}$$

$$\begin{aligned}\text{OFF-FLAVOR} = 2.02078 &+ .000002 \text{ (temperature x time x peroxide value)}\\ &+ .00374 \text{ (peroxide value)}^2\\ &+ .00006 \text{ (temperature x time)}\\ &\pm .44837\end{aligned}$$

MOUTH-FEEL = Not predictable

2. EQUATIONS PREDICTING ORGANOLEPTIC RESPONSES OF THE PANEL TO CANNED MARGARINE AS A FUNCTION OF TEMPERATURE (°F.), TIME (DAYS), PEROXIDE VALUES AND AOM VALUES

Note: The peroxide value and AOM values were derived from the oils separated from the canned margarine before storage.

$$
\begin{aligned}
\text{COLOR} = 1.24014 &+ .000006 \ (\text{temperature} \times \text{time} \times \text{peroxide value}) \\
&+ .00576 \ (\text{peroxide value})^2 \\
&+ .00034 \ (\text{temperature})^2 \\
&- .00028 \ (\text{temperature} \times \text{AOM value}) \\
&\pm .78447
\end{aligned}
$$

$$
\begin{aligned}
\text{ODOR} = 4.61219 &+ .000009 \ (\text{temperature} \times \text{time} \times \text{peroxide value}) \\
&- .04093 \ (\text{AOM Value}) \\
&+ .00042 \ (\text{temperature} \times \text{peroxide value}) \\
&+ .00006 \ (\text{time})^2 \\
&- .01076 \ (\text{time}) \\
&\pm .45837
\end{aligned}
$$

$$
\begin{aligned}
\text{OFF-FLAVOR} = 2.17110 &+ .000007 \ (\text{temperature} \times \text{time} \times \text{peroxide value}) \\
&+ .00429 \ (\text{peroxide value})^2 \\
&+ .00006 \ (\text{temperature} \times \text{time}) \\
&- .00002 \ (\text{time} \times \text{AOM value}) \\
&\pm .43801
\end{aligned}
$$

MOUTH-FEEL = Not predictable.

To evaluate the shelf life prediction equations, a set of values was computed by using the data obtained in this storage test study of canned margarine. The results are presented in Table IX. As may be seen it is possible to predict the shelf life of canned margarine with reasonable accuracy.

Another way of using the obtained prediction equations is to assign constant values for the temperature, and AOM to be inserted in the equations. In this manner the theoretical development of color, odor and off flavor can be computed as presented in graphical form in Figures 11 and 12.

During the study it was observed that the AOM stability test was not the most reliable for predicting the shelf life of the canned margarine. In spite of using four different methods for oil separation, only one method (thermal demulsification) was found which responded to off flavor, odor, and color changes in simultaneous correlation with the AOM stability and peroxide value.

The level of oxidized oil which determined the AOM and PV level of the initial oil was most meaningful for the development of rancidity as measured by chemical and sensory analysis. The temperature affected the sensory evaluation more than the chemical analysis.

Generally speaking, the canned margarine was very stable against chemically measured changes. At -20°F. hardly any changes were observed.

The mouth feel of this type of margarine is substantially different from our regular table grade product. This difference was so great that difficulty was encountered in making the sensory evaluation. The product does not have the customary flavor release.

The greatest physical change was observed in the fading of the color at the elevated storage temperature revealing the destruction of the used coloring material.

Since it was observed that the oxidation stability of the margarine in sealed cans was very high, it would be possible to package regular table grade margarine in a similar manner for selected military purposes.

TABLE V

PEROXIDE VALUE OF OILS SEPARATED FROM

CANNED MARGARINE

(THERMAL DEMULSIFICATION)

% Oxidized Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Initial	14.2	14.2	8.8	8.8	7.8	7.8	2.2	2.2	2.6	2.6
1 Month	15.8	14.2	10.8	9.2	10.6	8.0	5.0	2.6	4.6	2.8
2 Months	14.8	12.8	10.8	9.0	11.0	8.0	6.0	3.6	4.8	4.4
3 Months	15.6	14.2	10.0	9.2	11.6	8.8	8.2	3.4	5.0	3.6
4 Months	14.0	13.4	9.8	9.2	10.4	8.2	5.6	3.0	7.4	3.0
5 Months	16.2	14.0	10.0	9.8	14.4	8.8	6.6	3.8	5.2	3.4
6 Months	8.4	17.2	9.2	12.0	9.4	9.6	7.4	5.2	5.0	5.4

(SOLVENT EXTRACTION)

Initial	12.4	12.4	8.4	8.4	7.6	7.6	2.0	2.0	2.2	2.2
1 Month	14.8	14.2	10.4	8.6	13.8	8.2	5.2	2.0	4.8	2.8
2 Months	12.2	14.4	9.6	9.0	9.6	7.2	6.0	2.0	4.2	2.4
3 Months	10.6	12.8	8.4	8.4	9.8	7.4	6.0	3.6	3.8	1.8
4 Months	11.2	12.4	10.2	8.2	7.8	7.0	6.2	2.4	5.8	2.2
5 Months	12.4	13.6	7.8	8.8	9.8	8.2	3.6	2.4	2.4	2.8
6 Months	5.2	12.0	5.2	8.4	7.4	7.2	1	1.2	0.6	1.4

TABLE V (Continued)

(VACUUM DISTILLATION)

% Oxid. Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Initial	16.0	16.0	6.0	6.0	8.0	8.0	2.0	2.0	3.6	3.6
1 Month	15.8	13.1	11.6	9.2	10.8	8.7	5.2	1.8	5.3	3.5
2 Months	10.4	13.0	9.4	8.4	9.2	8.0	4.8	2.0	4.2	2.6
3 Months	12.2	12.4	8.8	8.0	7.8	7.4	5.0	2.0	4.0	2.4
4 Months	11.8	12.4	10.2	8.0	8.8	7.4	5.6	2.0	6.4	2.2
5 Months	11.4	13.4	7.2	8.4	11.0	8.2	3.6	3.2	2.4	3.2
6 Months	8.8	18.0	8.4	9.4	8.0	7.0	3.0	1.6	2.6	2.0

(CHEMICAL DEMULSIFICATION)

Initial	7.8	7.8	4.4	4.4	3.6	3.6	2.2	2.2	2.0	2.0
1 Month	7.6	6.4	5.4	5.0	5.4	3.7	2.0	1.7	2.2	2.2
2 Months	8.6	7.6	6.0	4.8	6.4	4.2	2.0	1.2	2.4	2.0
3 Months	5.2	6.2	4.4	5.6	3.0	4.0	1.6	1.2	1.8	1.8
4 Months	6.6	4.8	6.4	3.1	6.8	3.2	2.6	1.0	2.4	1.3
5 Months	4.2	6.8	3.4	5.0	3.0	4.8	0.6	1.0	0.8	1.8
6 Months	3.8	4.4	3.4	3.0	3.8	3.4	1.0	1.2	1.0	1.0

TABLE VI

AOM VALUE OF OIL SEPARATED FROM

CANNED MARGARINE

(THERMAL DEMULSIFICATION)

% Oxid. Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Initial	60	60	67	67	68	68	62	62	63	63
1 Month	53	59	64	69	67	69	57	69	61	70
2 Months	54	62	66	71	59	67	55	69	62	63
3 Months	54	62	79	69	64	71	61	58	85	69
4 Months	54	44	64	65	64	65	58	67	61	70
5 Months	58	61	69	71	57	68	60	65	68	63
6 Months	70	55	70	68	70	67	55	66	70	62
(SOLVENT EXTRACTION)										
Initial	56	56	67	67	53	53	49	49	46	46
1 Month	51	55	58	63	58	61	55	62	53	61
2 Months	68	62	86	70	53	66	54	64	52	52
3 Months	57	59	61	71	66	66	51	65	48	61
4 Months	66	60	80	68	63	69	56	64	56	62
5 Months	70	56	90	68	64	70	56	61	55	44
6 Months	78	53	61	68	60	71	34	60	39	52

TABLE VI (Continued)

(VACUUM DISTILLATION)

% Oxid. Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Initial	50	50	44	44	53	53	55	55	51	51
1 Month	22	57	57	62	57	66	53	63	68	61
2 Months	41	54	60	61	57	62	52	58	53	55
3 Months	51	54	60	62	63	67	55	64	58	65
4 Months	64	57	61	64	81	61	56	60	53	60
5 Months	58	43	71	63	58	76	61	65	65	66
6 Months	76	60	102	82	92	85	65	63	67	62
(CHEMICAL DEMULSIFICATION)										
Initial	24	24	27	27	22	22	32	32	32	32
1 Month	41	12	52	41	21	38	38	41	31	35
2 Months	42	41	38	46	39	46	31	42	30	46
3 Months	17	26	30	42	25	48	26	24	15	27
4 Months	51	18	58	42	39	53	32	33	24	26
5 Months	33	41	55	62	55	46	31	27	30	23
6 Months	40	48	49	61	48	57	35	42	36	43

TABLE VII

SENSORY EVALUATION OF

CANNED MARGARINE

% Oxidized Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Color Difference	2.25	2.25	2.37	2.37	2.81	2.81	2.31	2.31	1.25	1.25
Odor Difference	2.81	2.81	2.18	2.18	1.94	1.94	2.31	2.31	2.00	2.00
Off-Flavor	2.94	2.94	2.37	2.37	2.25	2.25	2.25	2.25	2.37	2.37
Mouth Feel	1.19	1.19	1.12	1.12	1.31	1.31	1.25	1.25	1.13	1.13
1 MONTH STORAGE										
Color Difference	4.33	2.22	2.55	2.36	4.85	1.55	3.84	2.97	2.12	0.86
Odor Difference	2.32	2.34	1.49	1.94	1.68	1.22	2.22	1.41	1.84	1.03
Off-Flavor	3.34	2.47	2.68	2.44	2.57	1.86	3.04	2.47	2.15	1.47
Mouth Feel	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
2 MONTHS STORAGE										
Color Difference	4.96	2.64	2.24	3.80	3.77	2.08	2.08	2.61	3.08	1.56
Odor Difference	2.57	2.04	2.19	1.07	2.37	1.43	2.11	1.59	2.03	1.75
Off-Flavor	3.17	3.67	3.33	2.00	3.00	1.67	1.83	3.33	3.50	1.67
Mouth Feel	1.10	0.88	1.01	0.96	1.01	1.03	1.17	1.03	1.15	1.48
3 MONTHS STORAGE										
Color Difference	5.99	2.65	2.34	2.30	4.02	1.49	4.51	1.67	4.51	1.17
Odor Difference	3.64	1.07	2.05	1.98	1.74	1.77	1.40	1.05	1.89	1.20
Off-Flavor	4.20	2.86	3.39	2.70	3.33	2.33	2.18	1.70	2.94	1.18
Mouth Feel	1.16	1.24	1.69	1.33	1.77	1.57	1.26	1.16	1.84	0.98

TABLE VII (Continued)

4 MONTHS STORAGE

% Oxidized Oil	20%		12.4%		5.8%		3.1%		0%	
	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F	100°F	-20°F
Color Difference	5.77	2.32	1.97	2.34	3.75	2.18	1.68	1.95	2.82	1.21
Odor Difference	2.94	1.22	1.90	1.16	2.97	1.53	2.28	1.16	3.13	1.31
Off-Flavor	4.44	2.54	2.92	1.62	3.59	1.73	1.81	2.35	3.19	1.29
Mouth Feel	1.03	1.08	1.35	1.54	0.98	1.19	1.08	1.22	1.64	1.22
5 MONTHS STORAGE										
Color Difference	5.99	2.67	2.54	2.19	3.85	1.33	3.85	2.40	2.47	1.52
Odor Difference	3.46	1.45	2.84	2.24	2.15	1.42	2.47	2.22	1.84	1.40
Off-Flavor	4.22	2.04	3.57	2.35	3.37	1.81	3.13	2.37	2.56	1.58
Mouth Feel	0.99	1.00	1.00	1.16	1.00	1.00	1.00	1.00	0.99	0.99
6 MONTHS STORAGE										
Color Difference	5.96	2.67	3.36	3.12	3.93	1.35	3.72	1.25	3.22	1.24
Odor Difference	4.05	2.04	3.02	2.62	2.74	1.67	2.58	0.96	2.06	1.58
Off-Flavor	4.62	2.50	3.57	2.22	2.99	1.92	3.55	1.33	3.57	1.55
Mouth Feel	1.18	1.09	1.40	0.98	1.34	1.06	1.30	1.07	1.33	1.57

TABLE VIII

CORRELATION COEFFICIENTS

	TEMP F°	TIME	% OIL	COLOR	ODOR	OFF FLAVOR	MOUTH FEEL	THERMAL P.V.	THERMAL AOM	SOLVENT P.V.	SOLVENT AOM	VACUUM P.V.	VACUUM AOM	CHEMICAL P.V.	CHEMICAL AOM
Temp.F°	1.00*	-.12	-.04	.56*	.52*	.62*	.16	.14	-.16	.08	-.15	.09	-.12	.09	-.17
Time		1.00*	.02	.13	.21	.12	.10	.12	.03	-.14	.17	-.05	.58*	-.24*	.40*
% Oxid.Oil			1.00	.34*	.28*	.43*	-.17	.82*	-.25*	.80*	.35*	.84*	-.13	.73	.10
Color				1.00*	.43*	.75*	.00	.48*	.20	.35*	.04	.34*	-.05	.27*	-.05
Odor					1.00*	.61*	-.00	.30*	-.16	.10	.06	.21	.10	.19	.06
Off-Flavor						1.00*	.04	.47*	-.16	.32*	.12	.38*	.02	.34*	.04
Mouthfeel							1.00*	-.12	.32*	-.19	-.16	-.17	.06	-.20	-.16
Thermal P.V.								1.00*	-.37*	.91*	.28*	.93*	-.14	.80*	.16
Thermal AOM									1.00*	-.32*	.22	-.33*	.38*	-.22	.12
Solvent P.V.										1.00*	.33*	.94*	-.27*	.88*	.05
Solvent AOM											1.00*	.23*	.26*	.32*	.45*
Vacuum P.V.												1.00*	-.18	.86*	.12
Vacuum AOM													1.00*	-.25*	.38*
Chemical P.V.														1.00*	.13
Chemical AOM															1.00*

1.000 = Perfect Correlation
*.23 or above = A Significant Correlation, with 69DF (α=0.05)

(G. W. Snedecor and W. G. Cochran, "Statistical Methods," Table All, p. 557, 1967)

TABLE IX

PREDICTIONS BASED UPON INITIAL OIL

EQUATION 1

Temp. °F	Time, Days	P.V.	AOM	Color Predicted	Color Actual	Odor Predicted	Odor Actual	Flavor Predicted	Flavor Actual
100	60	15.2	64	4.249	4.96	2.416	2.57	3.4273	3.17
100	60	11.0	76	3.5799	2.24	2.1811	2.19	2.9653	3.33
100	60	7.6	87	3.1598	3.77	1.9907	2.37	2.6880	3.00
100	60	2.6	121	3.3123	2.08	1.7107	2.11	2.437	1.83
100	60	2.4	96	2.7556	3.08	1.6995	2.03	2.4311	3.50
100	150	15.2	64	4.921	5.99	2.991	3.46	4.2409	4.22
100	150	11.0	76	3.9759	2.54	2.6428	2.84	3.7033	3.59
100	150	7.6	87	3.4334	3.85	2.3606	2.15	3.3648	3.37
100	150	2.6	121	3.4059	3.85	1.9456	2.47	3.0241	3.13
100	150	2.4	96	2.8204	2.47	1.9290	1.84	3.0143	2.56
-20	60	15.2	64	2.85197	2.64	1.39489	2.04	2.7764	3.67
-20	60	11.0	76	2.1279	3.80	1.4419	1.07	2.3749	2.00
-20	60	7.6	87	1.8057	2.08	1.4800	1.43	2.1466	1.67
-20	60	2.6	121	2.1022	2.61	1.5360	1.59	1.967	3.33
-20	60	2.4	96	1.551	1.56	1.5380	1.75	1.9645	1.67
-20	150	15.2	64	2.7425	2.67	1.4775	1.45	2.6137	2.04
-20	150	11.0	76	2.0488	2.19	1.5472	2.24	2.2273	2.35
-20	150	7.6	87	1.7510	1.33	1.6037	1.42	2.0112	1.81
-20	150	2.6	121	2.0835	2.40	1.6867	2.22	1.8505	2.37
-20	150	2.4	96	1.5340	1.52	1.6899	1.40	1.8479	1.58

TABLE IX (Continued)

PREDICTIONS BASED UPON OIL SEPARATED FROM THE INITIAL MARGARINE

EQUATION 2

Temp. °F	Time, Days	P.V.	AOM	Color Predicted	Color Actual	Odor Predicted	Odor Actual	Flavor Predicted	Flavor Actual
100	60	14.2	60	4.6327	4.96	3.0899	2.57	3.9205	3.17
100	60	8.8	67	3.5269	2.24	2.2851	2.19	3.1525	3.33
100	60	7.8	68	3.3673	3.77	2.1482	2.37	3.0381	3.00
100	60	2.2	62	3.0112	2.08	1.8561	2.11	2.5698	1.83
100	60	2.6	63	3.0087	3.08	1.8536	2.03	2.5637	3.50
100	150	14.2	60	5.3996	5.99	4.4057	3.46	5.2471	4.22
100	150	8.8	67	4.0022	2.54	3.1635	2.84	4.1263	3.59
100	150	7.8	68	3.7886	3.85	2.9455	2.15	3.9471	3.37
100	150	2.2	62	3.1300	3.85	2.1999	2.47	3.1369	3.13
100	150	2.6	63	3.14907	2.47	2.2298	1.84	3.1841	2.56
-20	60	14.2	60	2.7713	2.64	1.4542	2.04	2.7728	3.67
-20	60	8.8	67	2.1340	3.80	1.2713	1.07	2.2769	2.00
-20	60	7.8	68	2.051	2.08	1.2496	1.43	2.2129	1.67
-20	60	2.2	62	1.7353	2.61	1.6027	1.59	2.0269	3.33
-20	60	2.6	63	1.7492	1.56	1.6769	1.75	2.0343	1.67
-20	150	14.2	60	2.6179	2.67	1.3897	1.45	2.3779	2.04
-20	150	8.8	67	2.0389	2.19	1.2944	2.24	1.9375	2.35
-20	150	7.8	68	1.9669	1.33	1.2888	1.42	1.8843	1.81
-20	150	2.2	62	1.71161	2.40	1.7326	2.22	1.7797	2.37
-20	150	2.6	63	1.72107	1.52	1.6775	1.40	1.7765	1.58

FIGURE I

PEROXIDE VALUES OF OIL SEPARATED FROM CANNED MARGARINE
STORED AT 100° F

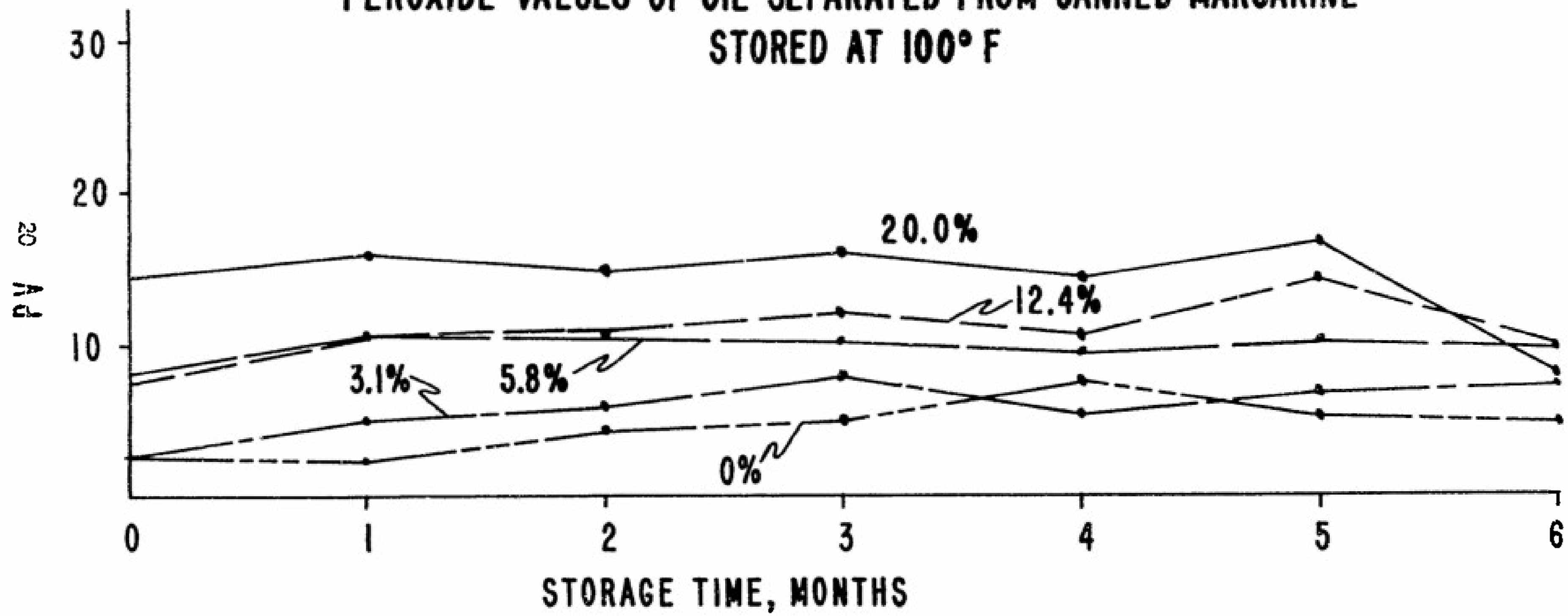


FIGURE 2

PEROXIDE VALUES OF OIL SEPARATED FROM CANNED MARGARINE
STORED AT −20° F

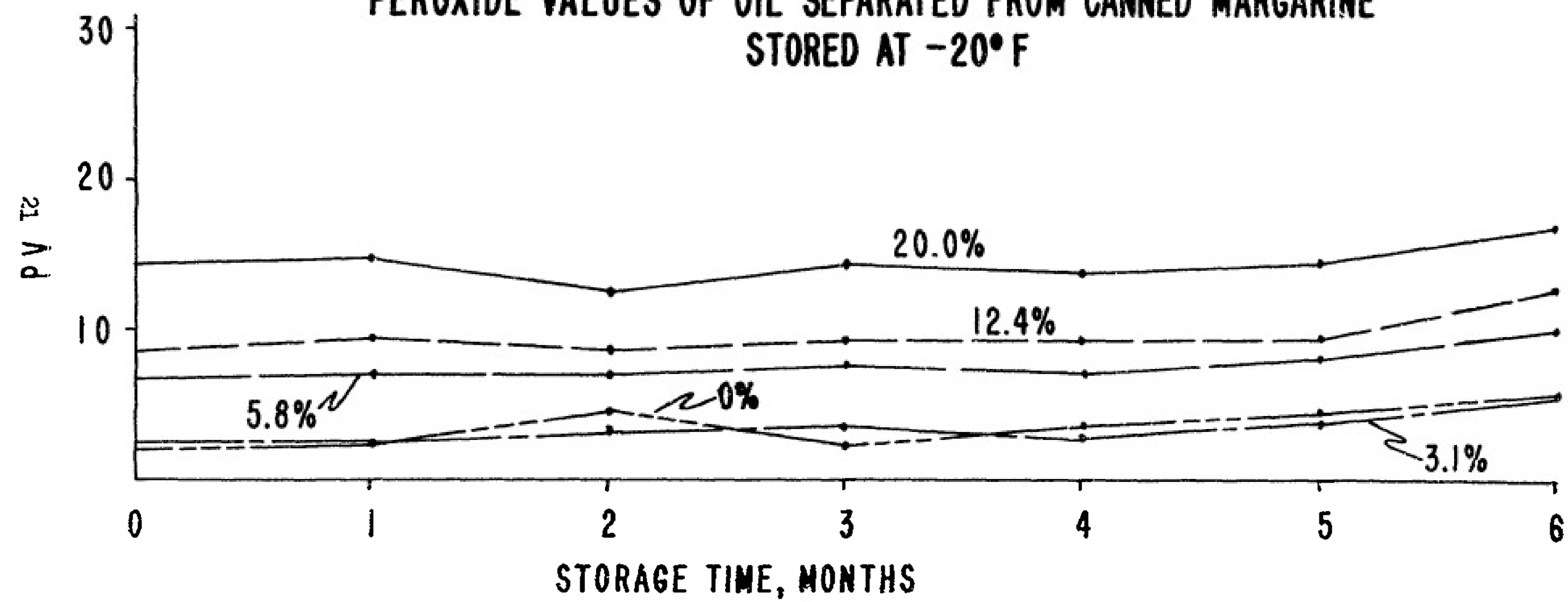


FIGURE 3

ACTIVE OXYGEN METHOD VALUES OF OIL SEPARATED FROM CANNED MARGARINE STORED AT 100° F

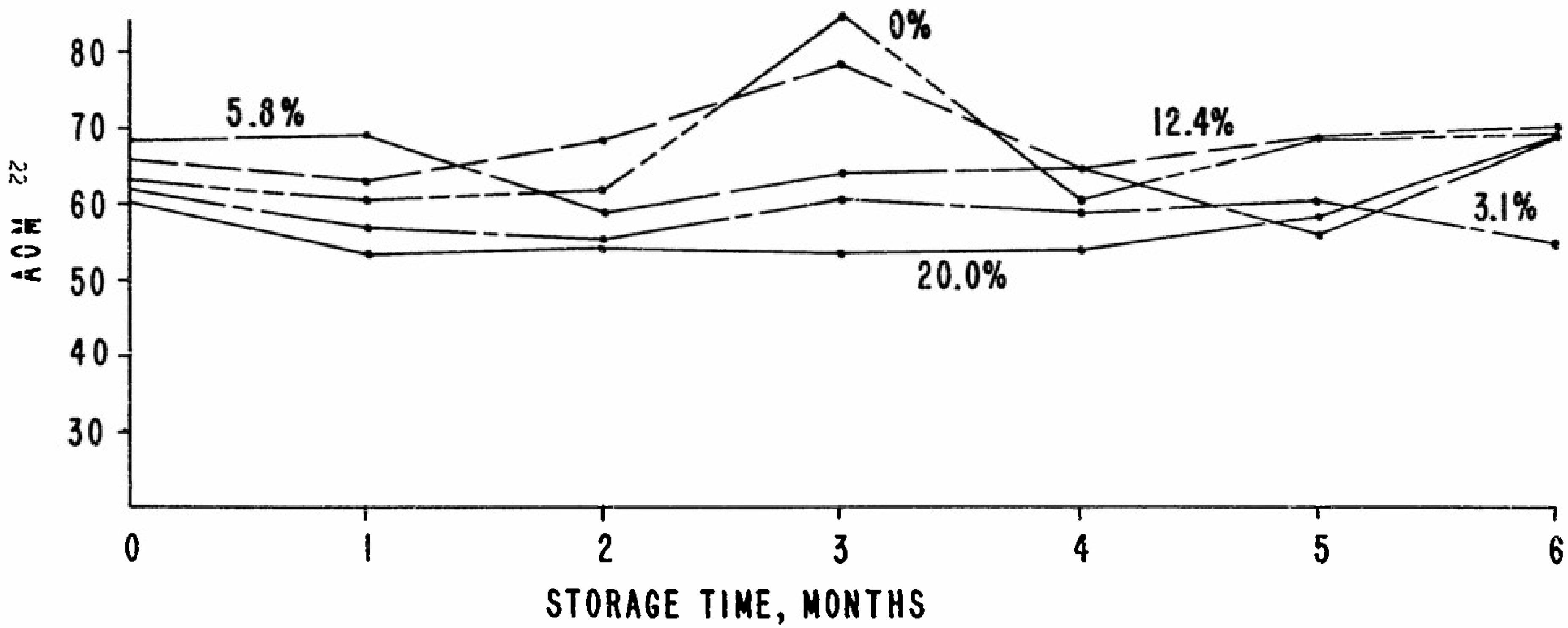


FIGURE 4

ACTIVE OXYGEN METHOD VALUES OF OIL SEPARATED FROM CANNED MARGARINE STORED AT −20° F

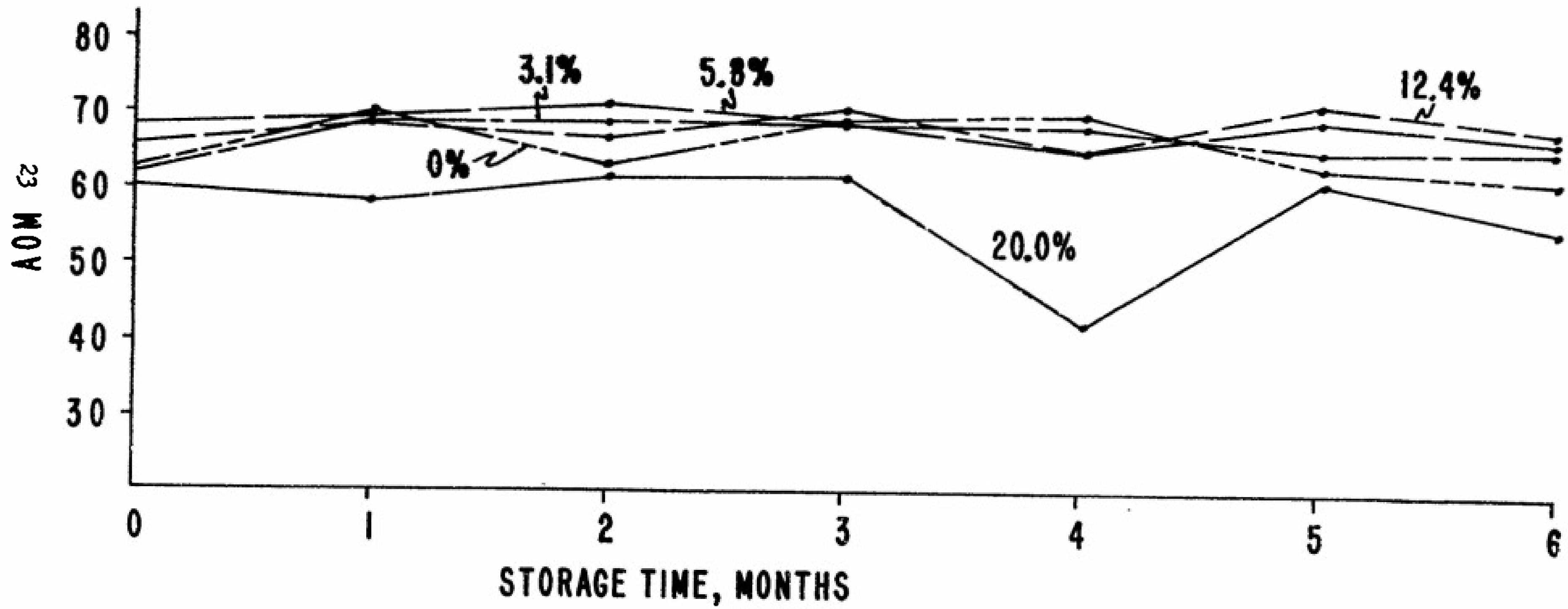


FIGURE 5

COLOR DIFFERENCE OF CANNED MARGARINE STORED AT 100° F

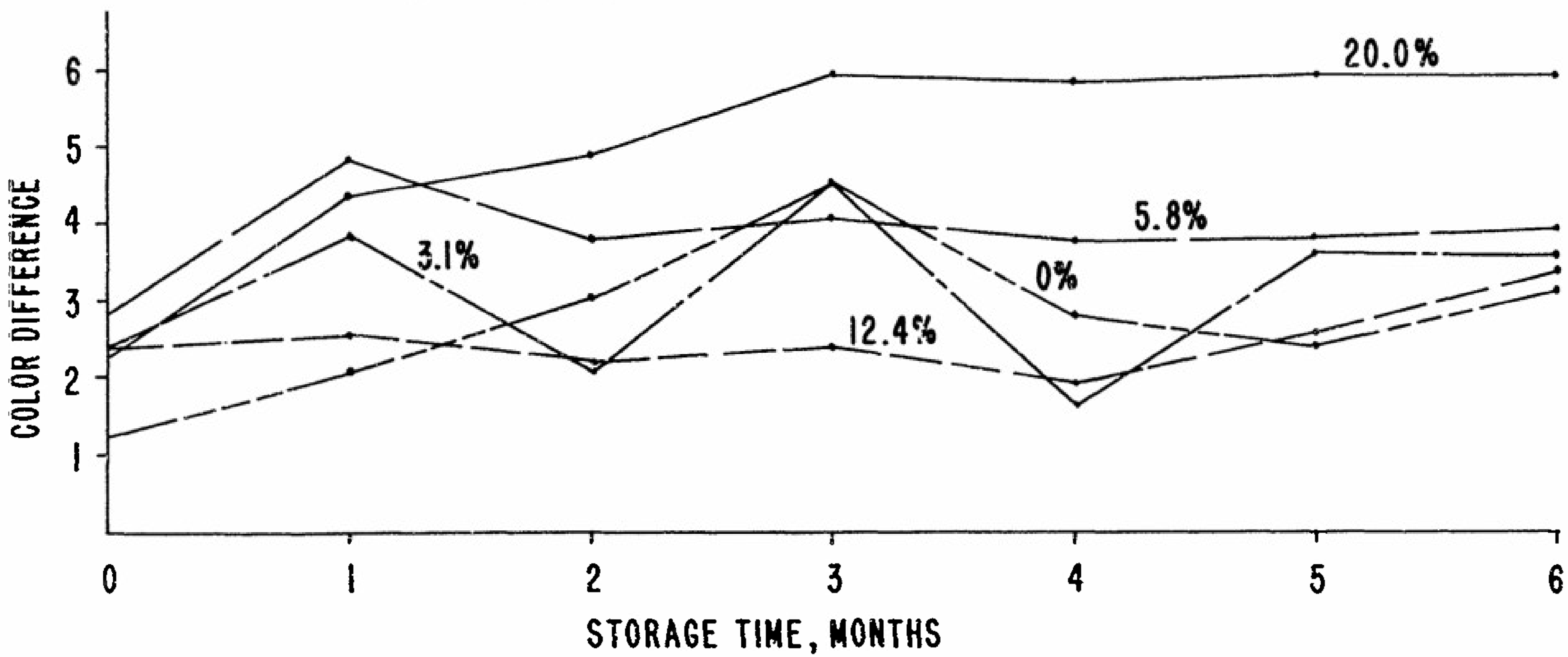


FIGURE 6

COLOR DIFFERENCE OF CANNED MARGARINE STORED AT −20° F

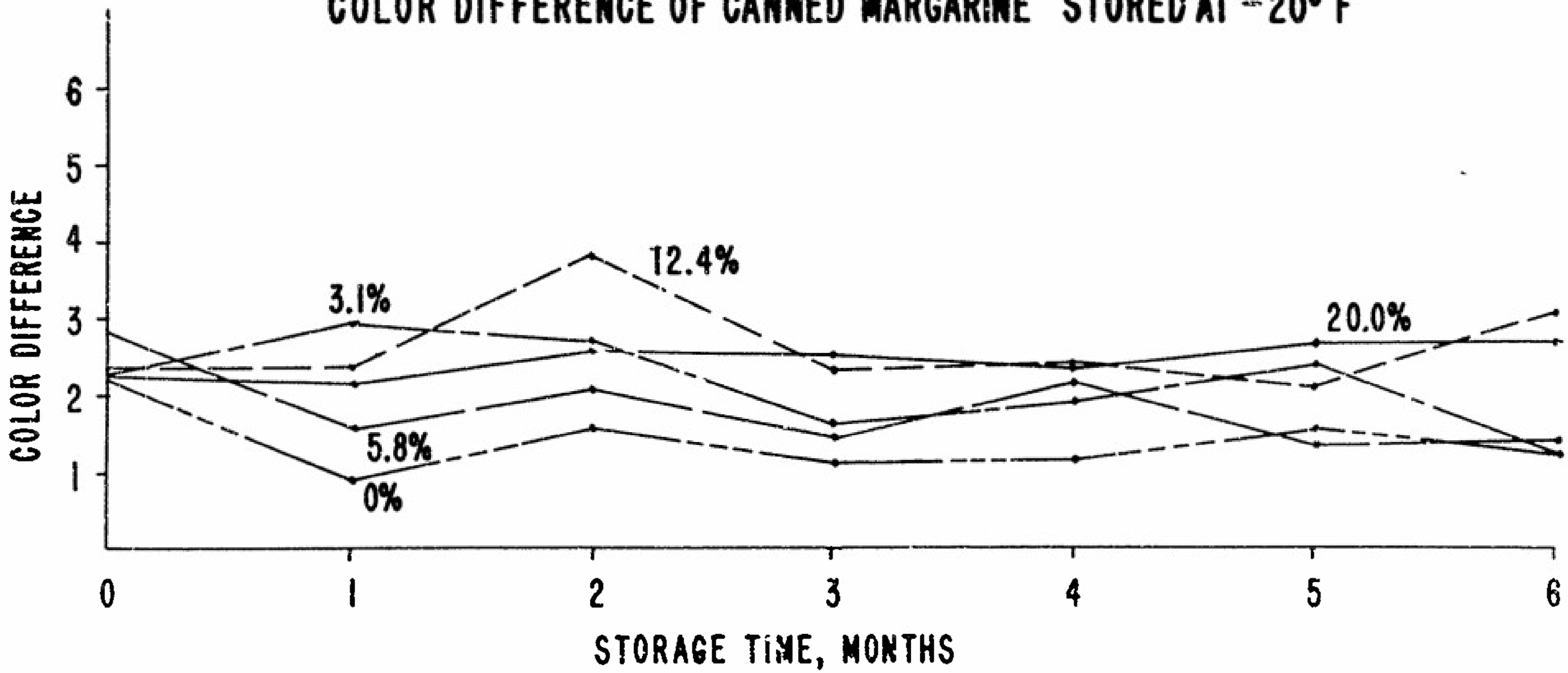


FIGURE 7

ODOR DIFFERENCE OF CANNED MARGARINE STORED AT 100° F

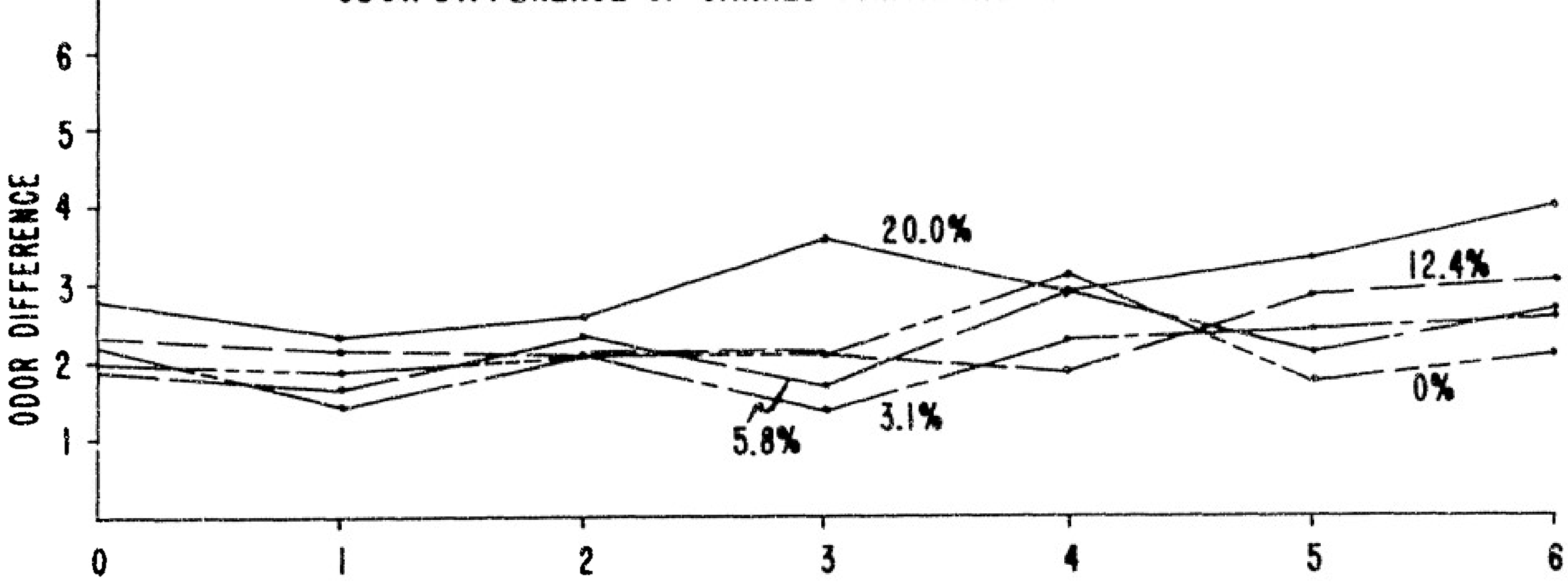


FIGURE 8

ODOR DIFFERENCE OF CANNED MARGARINE STORED AT -20° F

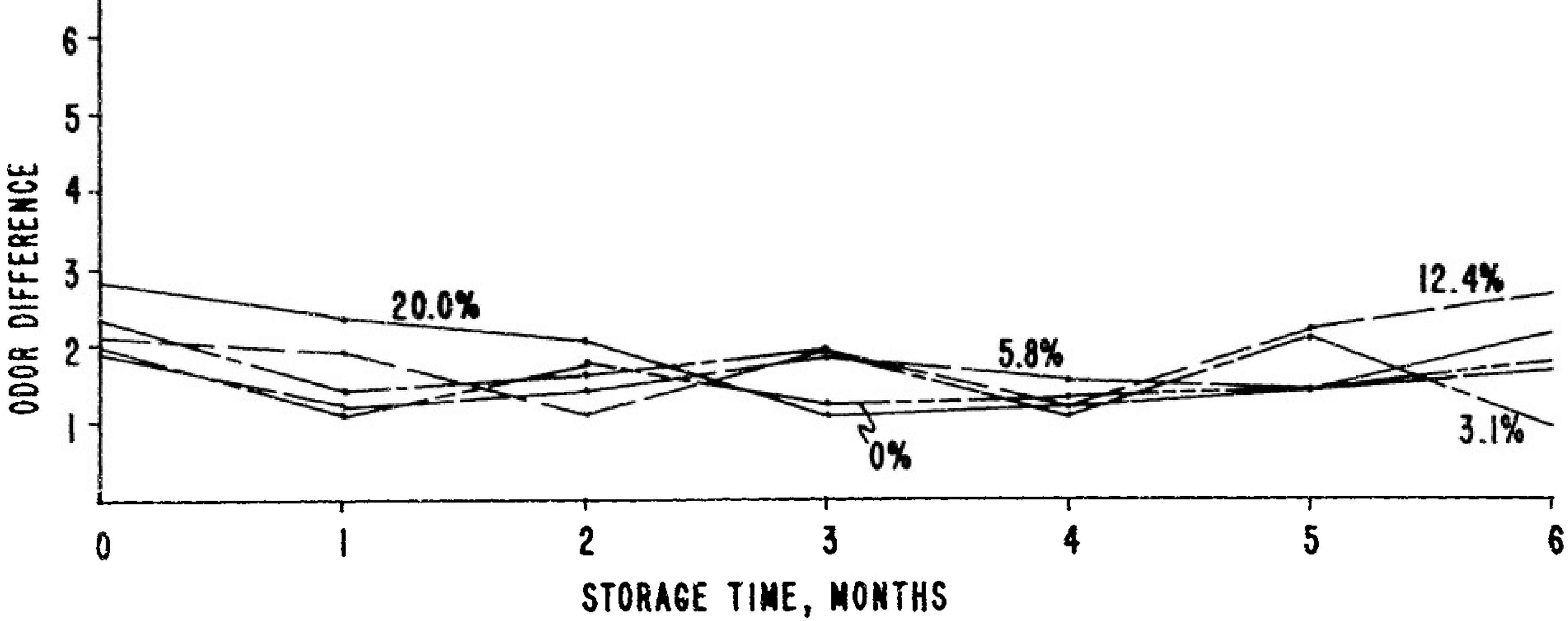


FIGURE 9

OFF-FLAVOR DIFFERENCE OF CANNED MARGARINE STORED AT 100° F

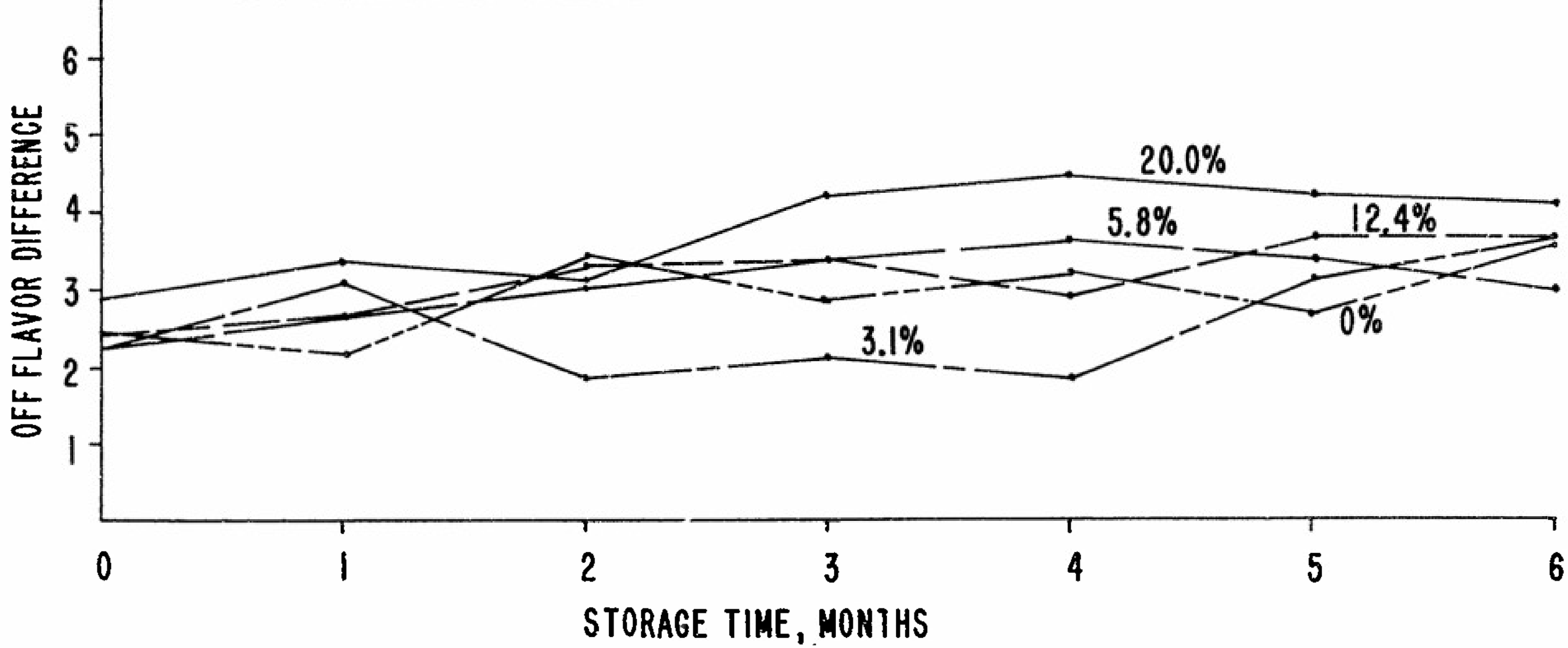


FIGURE 10

OFF-FLAVOR DIFFERENCE OF CANNED MARGARINE STORED AT −20° F

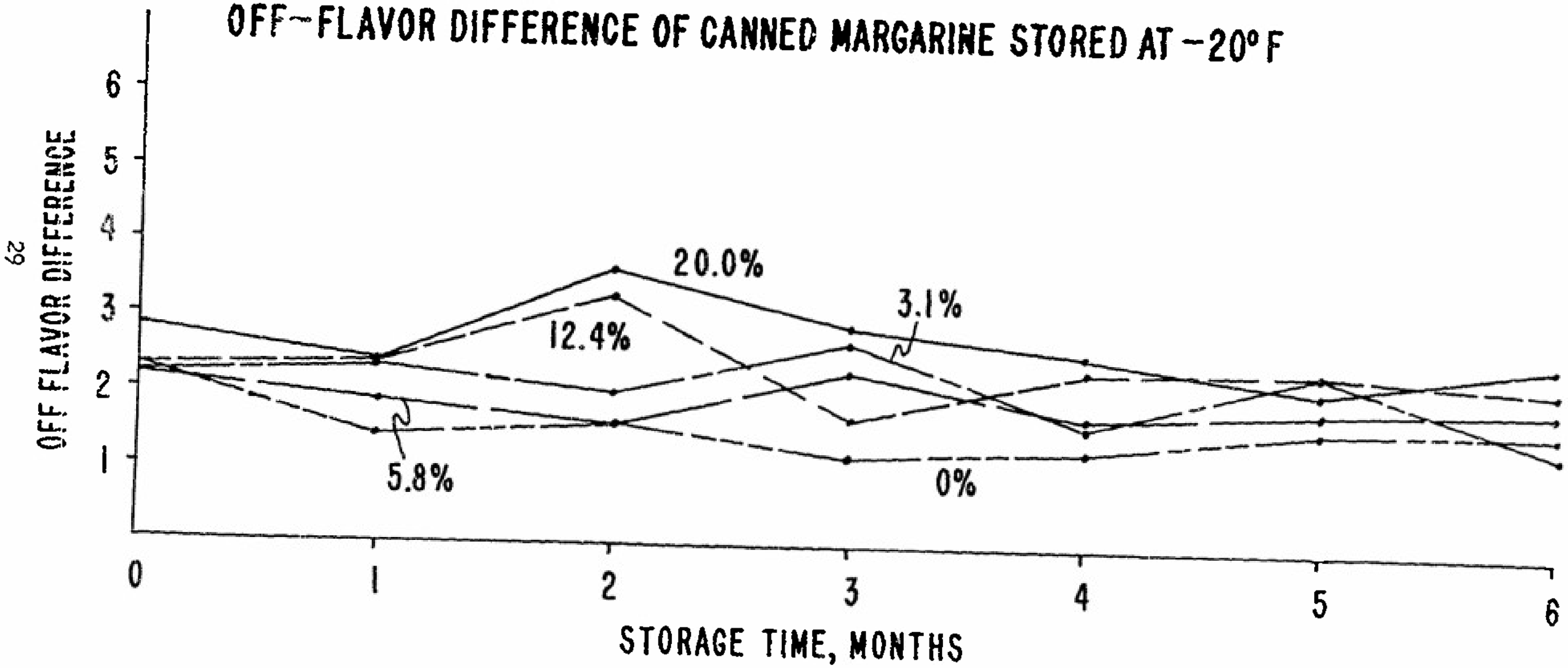
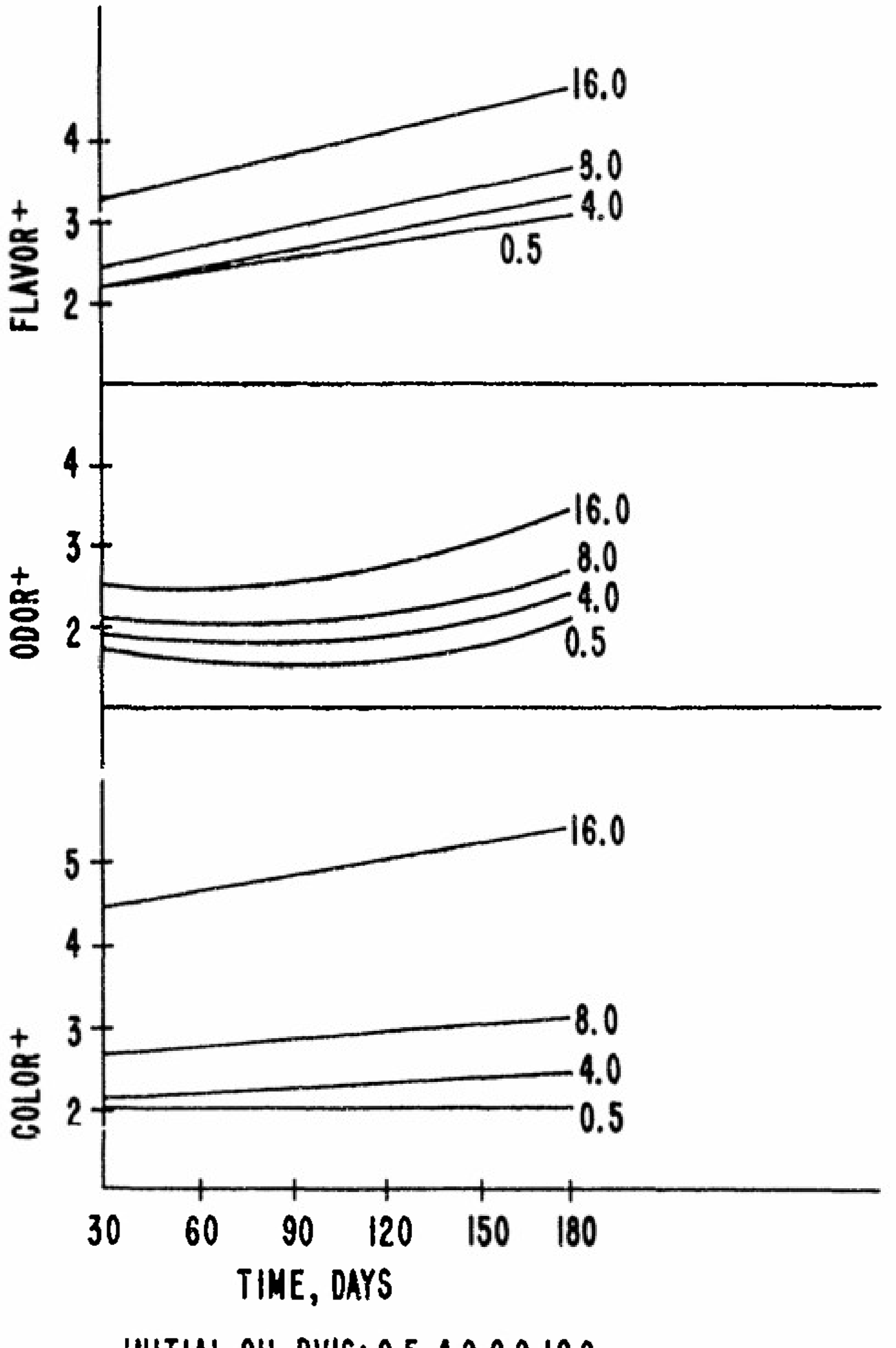


FIGURE II

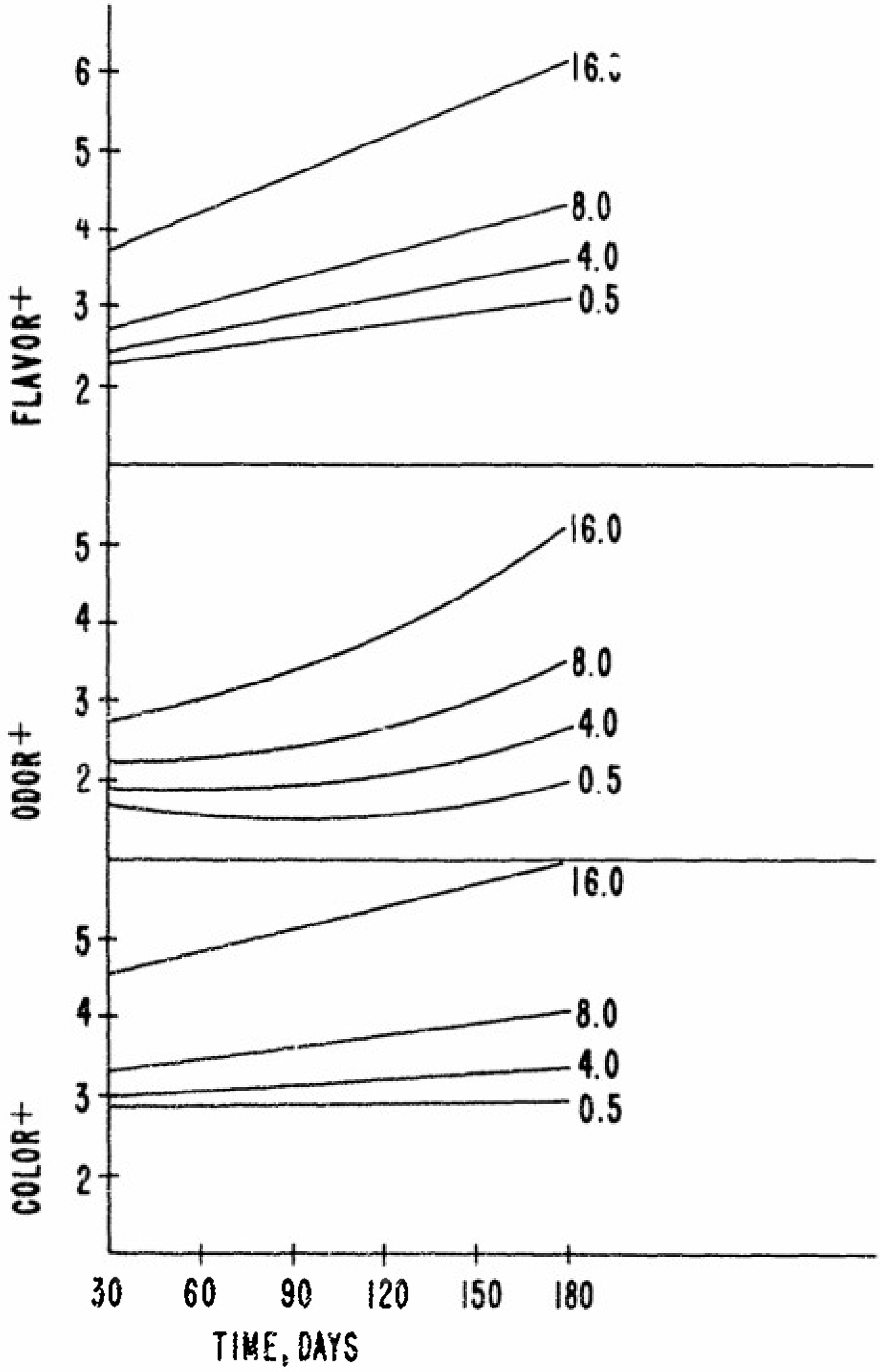
THEORETICAL SHELF LIFE OF CANNED MARGARINE
TEMPERATURE 100°F, AOM 65, EQUATION I



INITIAL OIL PV'S: 0.5, 4.0, 8.0, 16.0
+1.00 - NO DIFF. FROM THE STANDARD
6.00 - VERY MUCH DIFFERENCE

FIGURE 12

THEORETICAL SHELF LIFE OF CANNED MARGARINE, TEMPERATURE 100°F, AOM 65, EQUATION 2



OIL SEPARATED FROM INITAL MARG.
PV'S · 0.5, 4.0, 8.0, 16.0
+1.00 = NO DIFF. FROM THE STANDARD
6.00 = VERY MUCH DIFFERENCE